

Determination of crystallite size in Pt/C catalysts using benchtop ARL EQUINOX 100 XRD

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Introduction

The production of about 90 vol% of worlds chemicals is related to heterogeneous catalysis, which is crucial to reduce the amount of energy required for the process or to guide a chemical reaction to the desired product. Most important in heterogeneous catalysis is the active surface of the catalyst to get the best efficiency. Therefore, activated base materials (usually carbon or ceramics with large surface area) are impregnated with the active compound (nanoparticles of metals). One measure for the quality of the catalyst is the size of the nanoparticles especially after recycling of the materials. The most commonly used method to determine the crystallite size of nanoparticles is X-ray diffraction (XRD) where the peak width of reflections is directly related to the size of the scattering domain through Scherrer's equation. Therefore, it is possible to determine the crystallite size (CS) by determining the width of reflection in the diffraction pattern.



Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 X-ray Diffractometer employs a custom-designed Cu (50 W) micro-focus tube with mirror optics for high flux, which does not require external water chilling.

The ARL EQUINOX 100 Diffractometer provides very fast data collection times thanks to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously. It is therefore well suited for both reflection and transmission measurements.

Experimental

For XRD measurements, Pt/C powder was measured 5 min in reflection using a zero background sample cup. Crystallite size determination (Williamson-Hall method) was carried out using MDI JADE 2010.

Results

The FWHM of the reflections was determined applying a Le Bail fit and afterwards calculating CS via Williamson-Hall method (cf. Figure 1) also taking into account strain and instrument broadening (determined from NIST660 LaB6). Refinements yield a CS of 2.5 nm, which is in very good agreement to literature values.

Conclusion

The benchtop ARL EQUINOX 100 X-ray Diffractometer in combination with the MDI JADE 2010 Software suite is a convenient solution to easily determine CS of Pt nanoparticles in Pt/C catalysts.

Figure 1. Diffraction pattern of Pt/C (green: Pt; blue: C); Williamson-Hall plot of Pt/C (Red area indicates 1.5 σ).

